

Gas "wets" a solid wall in orbit¹

John Hegseth², Yves Garrabos³, Vadim S. Nikolayev³ Carole Lecoutre-Chabot³,

Regis Wunenburger³, and Daniel Beysens³

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2 Department of Physics, University of New Orleans, New Orleans, Louisiana 70148, U.S.A. To whom correspondence should be addressed.

3 ESEME, Institut de Chimie de la Matière Condensée de Bordeaux, CNRS, Université de Bordeaux I, Avenue du Dr. Schweitzer, F-33608 Pessac Cedex, France.

ABSTRACT

When co-existing gas and liquid phases of pure fluid are heated through their critical point, large scale density fluctuations make the fluid extremely compressible, expandable, and slows the diffusive transport. These properties lead to perfect wetting by the liquid phase (zero contact-angle) near the critical temperature T_C . We have found experimentally, however, that when the system's temperature T is being increased to T_C , so that it's slightly out of equilibrium, the apparent contact-angle is very *large* (up to 110°), and the *gas* appears to “wet” the solid surface. These experiments were performed and repeated on several missions on the Mir space station using the Alice-II instrument, to suppress buoyancy driven flows and gravitational constraints on the liquid-gas interface. These unexpected results are robust, i.e., they are observed either under continuous heating (ramping) or stepping by positive temperature quenches, for various morphologies of the gas bubble and in different fluids (SF_6 and CO_2). We consider as possible causes of this phenomenon both a surface-tension gradient, due to a temperature gradient along the interface, and the vapor recoil force, due to evaporation. It appears that the vapor recoil force has a more dominant divergence and explains qualitatively the large apparent contact-angle far below T_C .

KEY WORDS: contact -angle, surface tension, critical exponent, thermal-capillary flow, Marangoni flow, principal axis, vapor recoil

INTRODUCTION

When a coexisting liquid-gas mixture of a single species fluid is heated into the gas phase a complex process of fluid dynamics, heat transfer, and interfacial phenomena usually occurs [1]. This process, that is often called boiling, is important in many applications because of the large heat transfer that it can facilitate so that many types of heat transfer technology use this process. Many of these complications are caused by the buoyancy from gravity that lift the gas bubbles that nucleate on a hot surface. Near the liquid-gas critical point material and thermal properties that play an important roll in the boiling process such as the surface tension vary considerably with temperature [2]. These properties vary according to well-known universal power laws that either converge or diverge (e.g., surface tension goes to zero) and lead to perfect wetting by the liquid phase (zero contact-angle) near the critical temperature T_C [3]. In a boiling process, we can expect a perfectly wetted wall to dry from evaporation resulting in liquid-gas-solid contact lines. The same physics that makes perfect wetting in equilibrium will result in a boundary condition of zero contact-angle when heat is applied. In the following we report on observations of a single bubble in a thin constant mass cell that is filled with fluid very close to its critical density. This thin cell produces a considerable constraint on the bubble and allows the entire bubble to be observed as the heat is applied. As the liquid-gas mixture is heated toward the critical point, the diverging and conversing material properties produce a large effect on the bubble shape.

RESULTS

In these experiments, a thin layer of SF_6 or CO_2 was sandwiched between two sapphire windows and surrounded by a copper housing in the optical cell shown in Figure 1. Figures 2 and 3 show the results from a typical run where the cell is heated linearly in time to a temperature greater than the critical temperature T_C , while the liquid-gas interface was visualized through light transmission normal to the windows. Figure 3 shows several cell

images obtained during the heating. Because the contact-angle is zero near the critical point, the liquid-gas meniscus between the two parallel windows forms a semi-circular interface in the plane perpendicular to the windows. The interface appears dark in the images because the liquid-gas meniscus refracts the normally incident light away from the cell axis. We have verified by a ray-tracing model that all of the normally incident light on the meniscus is refracted out of the field of view. This shows that in normally incident light, the dark region measures the radius of the semi-circular meniscus.

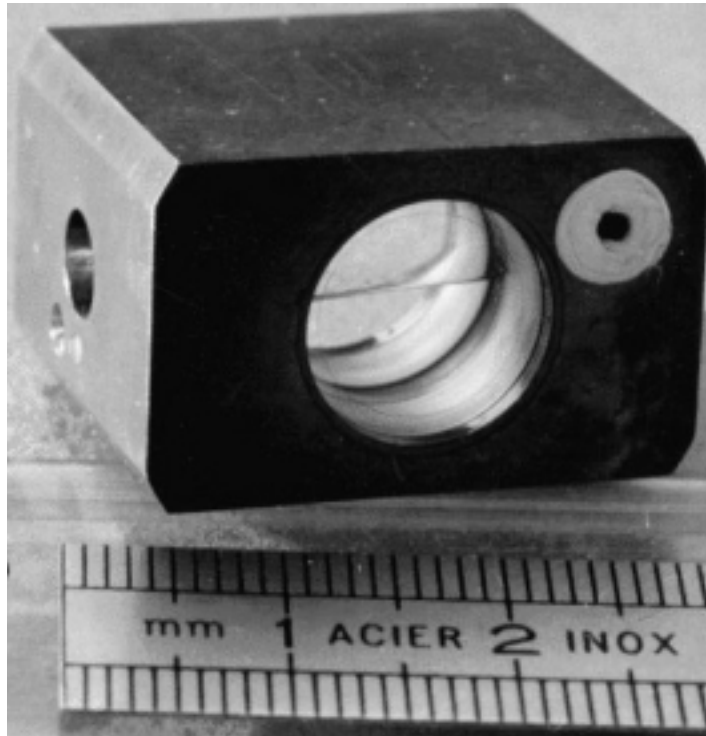


Figure 1. Sample cell. Shown is the sample cell in terrestrial gravity filled with SF_6 fluid. The meniscus between the co-existing liquid and gas phases below the critical point can be seen. The average density exceeds the critical density by $0.3\% \pm 0.01\%$. The fluid volume (12.000 mm diameter, 1.664 mm thickness) is contained between two sapphire windows and a CuBeCo alloy housing.

These results were obtained and repeated using several samples of both SF_6 and CO_2 at different cell aspect ratios and heating protocols (ramping rates and quenches) on several

French/Russian (Cassiopée and Pegase) and French/American (GMSF) missions on the Mir space station using the Alice-II instrument [4]. This instrument is specially designed to obtain high precision temperature control (stability of $\approx 10 \mu\text{K}$ over 50 hours, repeatability of $\approx 40 \mu\text{K}$ over 7 days). To place the samples near the critical point, constant mass cells are prepared with a high precision density, to 0.02%, by observing the volume fraction change of the cells as a function of temperature on the ground [5]. Two cylindrical sapphire windows 12mm in diameter and 90mm long are pressed into a copper block with a corresponding cylindrical hole and glued to the copper at the sides of the sapphire. This method avoids the unknown volume associated with o-rings, etc., allowing the above high precision density measurements to be verified. Similar ground based experiments were done before these experiments, yielding completely different results [5]. In this case the interface is horizontal except very near a wall.

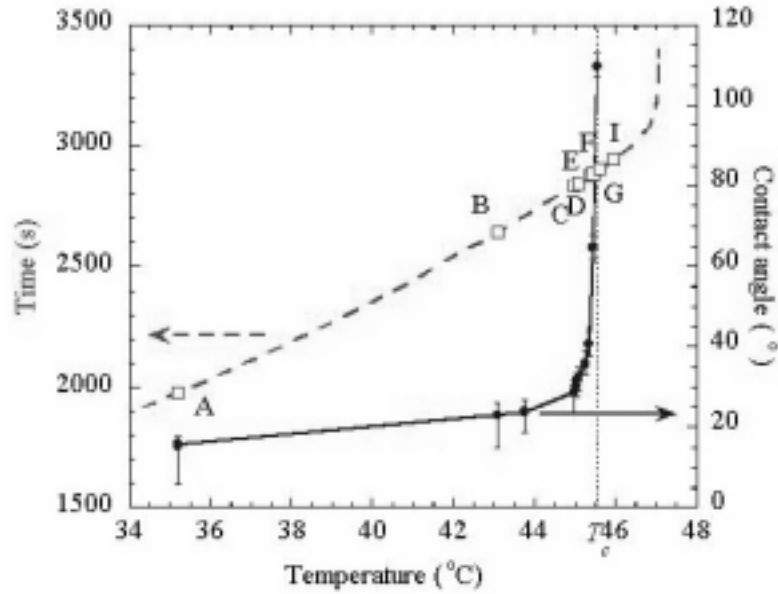


Figure 2. Plots of the contact angles as a function of temperature and the temperature of the cell as a function of time. The critical temperature of SF_6 is $T_c = 45.54^\circ\text{C}$ as shown. The mean value of dT/dt at T_c is about 8.4mK/s . Also shown are points where the images of Figure 3 were taken with the corresponding letter labels.

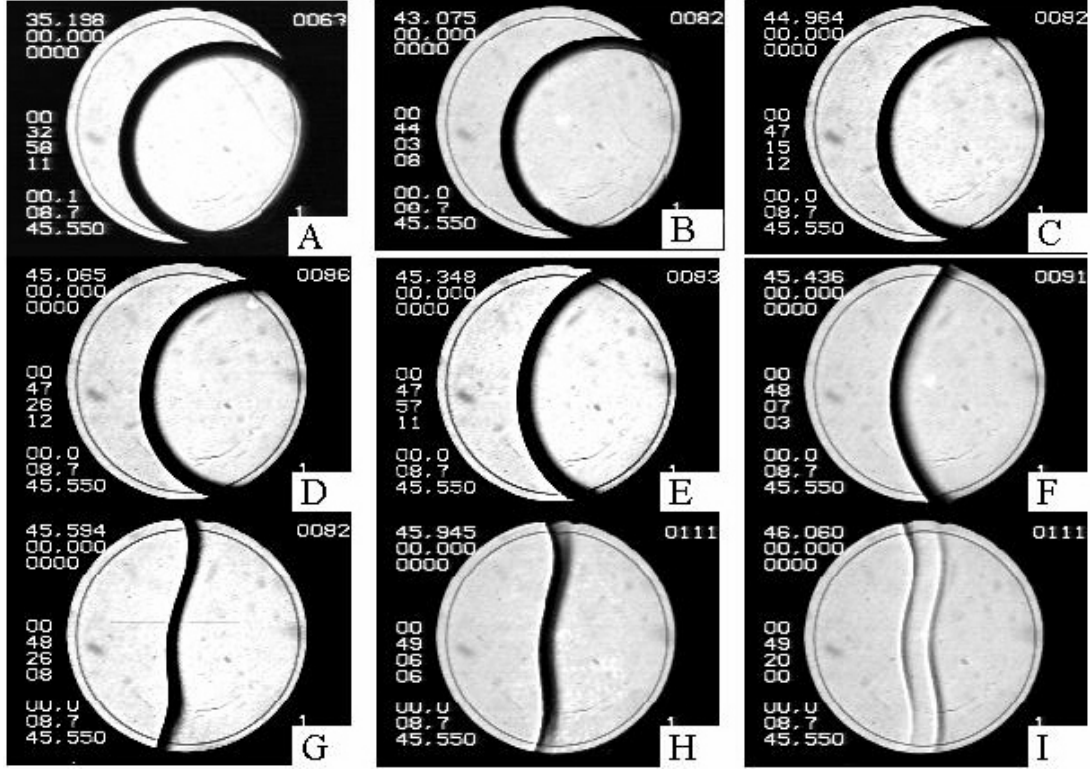


Figure 3. Contact angle and bubble shape. Shown is a series of images of the bubble at various temperatures indicated in Figure 2. The change in the contact angle and bubble shape is clearly seen. The first row of images shows that the bubble distortion and contact angle changes occur even far below the critical point. The second row of images illustrates the rapid changes that occur close to the critical point. The last row of images shows a density gradient that replaces the interface above the critical temperature.

In our system the liquid wets the solid, so that the initial state of our system before heating is a flat bubble constrained by the two windows and the cell edge. The initial off-center position of the bubble, with part of the bubble touching the copper ring, occur because the cell windows are not exactly parallel and constrain the bubble to press against the ring. We can estimate the tilt angle, θ , between the windows from the mechanical precision of the cell manufacturing process and it does not exceed 0.3° . In thicker cells when the bubbles are not pressed against the ring or when the bubble is constrained to not touch

the side-wall, no similar bubble deformation is observed.

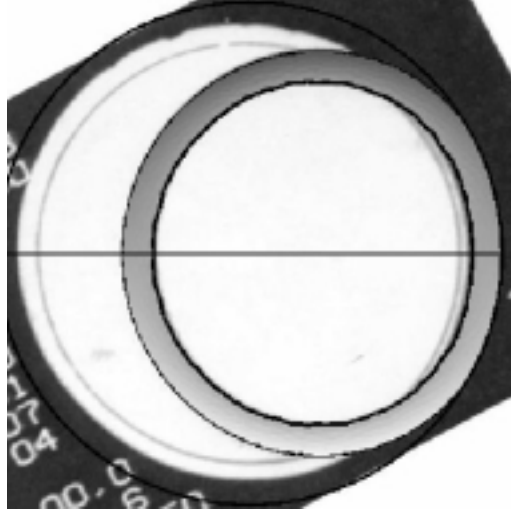
Figures 2 and 3 show that as the critical point is approached the gas region spreads along the copper side-wall. Because the curvature on a large portion of the bubble is constant, we can extrapolate it to the wall to define an apparent contact angle and this apparent contact-angle increases. The dark region that measures the meniscus radius does not appear to be significantly effected except at the side-wall where it disappears. Figures 2 and 3 show that there is a significant bubble deformation quite far below the critical temperature T_C . Near the critical temperature the gas has spread over nearly half the copper side-wall. The apparent contact-angle becomes so large that the gas phase appears to "wet" a large portion of the cell surface. When crossing the critical point, as shown in images D-H of Figure 3, the vapor bubble loses its convexity and rapidly evolves. At $T \approx T_C$, there is no latent heat and no surface tension so that the interface becomes a density gradient. This is shown in the last several images of Figure 3.

DISCUSSION

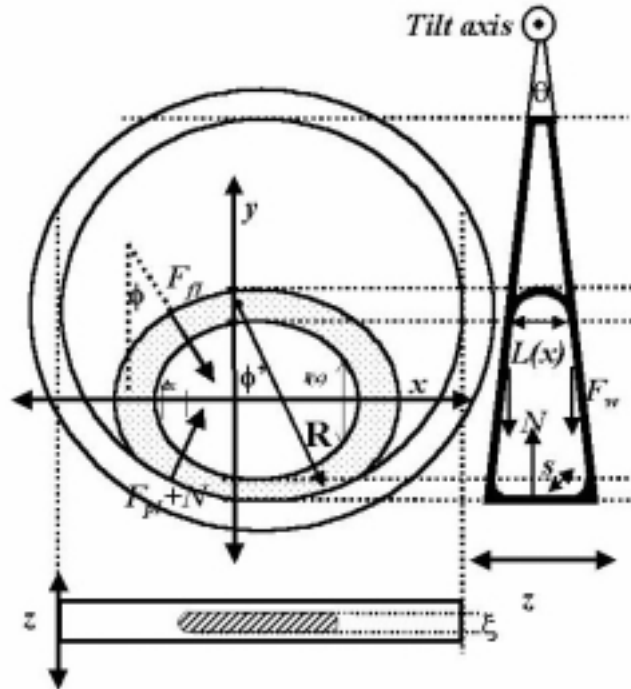
Figures 2 and 3 show that there is significant bubble deformation below T_C where the surface tension is still relatively large and the shape may be analyzed by quasi-static arguments. If a temperature change, δT , appears along the liquid-gas interface, it will create a surface tension gradient $\delta\sigma=(d\sigma/dT)\delta T$ that will drive a thermal-capillary or a Marangoni flow in the bulk of both fluids [6, 7, 8]. Such a flow could modify the shape of the bubble. We have previously seen Marangoni rolls in similar cells in the same apparatus in ground based experiments using the shadowgraph effect. These rolls have a horizontal width of the order of the cell thickness and are transient. They form near the gas-liquid interface after a large temperature quench and travel along the horizontal interface for several seconds before disappearing. One of us (J.H.) has also seen similar shadowgraph images of sustained Marangoni convection in evaporating methanol along a thin horizontal interface in similar

geometry verifying that the transient rolls are Marangoni rolls, probably also driven by evaporation. We have not seen any evidence of the steady convection that is required to create and maintain the observed bubble shape in our experiments. We have, however, on occasion seen transient plumbs in some experimental runs from bubble coalescence but these events only had a small transient influence on the bubble shape. The fact that there is no convection farther away from the critical point shows that the interface is isothermal. This may not necessarily be true closer to the critical point. The dimensionless parameter that governs the stability of an isothermal interface, in the presence of an externally imposed temperature gradient normal to the interface, is the Marangoni number given by $Ma = \delta\sigma L / (\eta D_{th})$. L is the characteristic length over the distance that σ varies by $\delta\sigma$, η , and D_{th} are the viscosity, and the thermal diffusivity respectively. This number is the ratio of the surface tension driving force to the viscous dissipation force so that if this number is larger than ~ 100 a stationary interface becomes unstable to surface tension driven flow. Close to the critical point the surface tension vanishes with the universal form $\sigma = \sigma_o (T_C - T)^{2\nu}$, where $\nu = 0.63$ is a universal exponent, $\delta\sigma = -2\nu\sigma_o (T_C - T)^{2\nu-1} \delta T(\mathbf{x}) \sim (T_C - T)^{0.26} \delta T(\mathbf{x})$. The D_{th} factor in the denominator of Ma disappears with the form $D_{th} \sim (T_C - T)^{0.85}$ [9] so that Ma has the form $Ma \sim (T_C - T)^{-0.59}$ that diverges as $T \rightarrow T_C$. We note, however, that we do not have an externally imposed temperature gradient that corresponds to the classical Marangoni instability problem. A temperature gradient across the interface driven by evaporative heat transfer would tend to equilibrate any $\delta T(\mathbf{x})$ to maintain a uniform saturation pressure in the cell, i.e., local temperature perturbations away from an isothermal interface would be strongly dampened by evaporation. Even far from saturation an evaporating interface tends to a uniform temperature through convective heat transport in the absence of an externally imposed temperature gradient, as observed in Reference [7]. In addition, the strength of any possible surface tension driven convection is measured by the characteristic velocity

$U \sim \delta\sigma/\eta$. This velocity goes to zero near T_c as $U \sim (T_c - T)^{0.26}$. Very close to the critical point, the velocity of the flow produced by a possible $\delta T(x)$ is probably too small to observe and certainly too small to drive the large bubble distortions that we have observed.



a



b

Figure 4. The bubble in equilibrium. a) Comparison of a 3-D numerical simulation of the equilibrium bubble shape with an actual image of the bubble. The window tilt angle input to the simulation is 0.3° . b) Diagram showing the forces on the bubble in equilibrium.

We next discuss the possible influence of the window tilt on the bubble shape. We first note that there are several important constraints present in our system. A liquid-gas mixture in coexistence has a specific volume fraction for gas given by the lever rule. The zero contact-angle results in principal axes of curvature that are parallel to the cylinder axis so that the edge of the bubble in Figure 3 appears to be a dark thick line. These principal axes are oriented in a direction normal to this thick line. These regions of curved liquid-gas interface not in contact with the wall have a Laplace pressure $p = \sigma c$ at each element of free interface. Because both p and σ are constant on the interface c is also constant at each point in this region. We have computed the initial equilibrium bubble shape by minimizing the system's free energy as shown in Figure 4a. This calculation used the Surface Evolver software that employs the finite element method. We used a zero contact angle and a window tilt of 0.3° . This calculation matches the initial bubble shape, verifies that the bubble is touching the side-wall, and verifies that the window tilts are the cause of this initial shape. To gain some qualitative understanding of this initial bubble shape we consider the forces on the bubble and the condition of mechanical equilibrium. The slight tilt of the windows about a tilt axis parallel to x by an angle θ makes a net reaction force from the windows in the $(-y)$ direction, as shown in Figure 4b. The rotational symmetry that the bubble would have with parallel windows is broken in to a discrete reflection symmetry as the bubble changes from a circular cross section in the (x, y) plane to an oval cross section. We first reduce this problem to 2-D by integrating along all of the principal axes of the interface parallel to z to get net forces in the (x, y) plane as shown in Figure 4. The y -axis is in the reflection symmetry plane of the bubble (also the plane of maximum tilt that defines θ) and all of the x -components of any normal or interfacial stress cancel by this reflection symmetry. At each element of the bubble between x and $x+dx$ there are several y -

components of force. There is the y-component of the free interface, $F_{\text{if}} \cos \phi$, that pushes in the (-y) direction, where $F_{\text{if}} = \sigma L$ and L is the distance between the windows that depends on the shape of the bubble, $y(x)$. The flat tilted windows also produce a force, F_w , that pushes in the (-y) direction that is equal to $\int p \sin(\theta/2) dy$. The limits depend on the shape of the region in contact with the window that is parallel to the y-axis, $l(x)$. Below the x-axis there are several forces that push the gas in the bubble in the (+y) direction. The part that is not pinned by the wall has a (+y)-component to $F_{\text{if}} \cos \phi$. The large curvature varies more strongly in this edge region of the bubble and the small curvature is also varying considerably in order to maintain the constant curvature condition. The part of the interface pinned by the wall has both normal and interfacial forces that all push toward the center of the cell. The dark line corresponding to the interface is concentric with the cell radius so its large curvature principal axis passes through the cylinder axis. The thickness of this dark line is less than it would be if it were not pinned by the side-wall implying that ξ , as defined in Figure 4b, is constant as we have also seen in the numerical simulation. This pinned interface produces a force F_{pl} of constant magnitude that points toward the cell's center at each dx . This force is $\sigma s \cos \phi'$, where s is the length associated with the integration and ϕ' is the angle between the cell radius and the y-axis. There is also a normal force $N = p \xi$ on the bubble from the side-wall wall region that is in contact with the bubble, as shown in Figure 4. Because the higher pressure in the bubble is caused by this Laplace pressure, $p = \sigma c$, all of the above forces are proportional to σ . All of these forces must also integrate to zero so that a constant σ on the isothermal interface factors out of the mechanical equilibrium condition and the bubble shape depends only on geometrical factors. The bubble shape should therefore also be independent of temperature. Although the window tilt and the constraining walls play an important roll in defining the boundary conditions and position

of the bubble they can not be the cause of the bubble deformation that we have observed. As $\sigma(T)$ decreases when the bubble is heated, all the forces on the bubble decrease proportionately, i.e., bubble become more easily deformed but the reaction forces from the window tilt also decreases as the Laplace force decreases. We also note that in some experiments where temperature was raised by steps and allowed to equilibrate between them, the bubble returned to its initial shape. We conclude that an external force is applied to this system as the heating is applied and the temperature is increased.

When very close to the critical point the vapor bubble loses its convexity and rapidly evolves, as shown in images D-H of Figure 3. As shown above, convective transport of heat can not equilibrate the interface temperature. The other possible modes of heat transport are also very inefficient as $T \rightarrow T_C$. Temperature diffusion becomes small because $D_{th} \sim (T_C - T)^{0.85} \rightarrow 0$ as $T \rightarrow T_C$, the latent heat also goes to zero as $(T_C - T)^\beta$ where $\beta = 0.325$. In our experiment the cell is heated past T_C , so the possibility of $\delta T(\mathbf{x})$ along the interface increases close to T_C . There is, however, a very efficient heat transfer process close to the critical point. This process is an adiabatic heat transfer process caused by the diverging compressibility and thermal expansion coefficient particular to near critical fluids [10]. The large thermal expansion and the slow diffusive transport of thermal energy in a near critical fluid lead to a low-density thermal boundary layer near the heating walls. These expanded boundary layers compress the bulk fluid, heating it adiabatically. In a liquid-gas mixture, the compression by the boundary layer may heat the gas more than the liquid, leading to a quite large temperature difference [11]. Recently, in fact, it has been observed that when a two-phase system's temperature is quenched upward, the gas temperature may actually exceed the wall temperature [12]. Close to the critical point, a temperature change, $\delta T(\mathbf{x})$, where \mathbf{x} is a position at the interface, could change the bubble's shape by producing surface tension change, $\delta\sigma$, on the interface. After the cell heating is started, the bubble deforms and

the Laplace formula becomes $p = \sigma(\mathbf{x})c(\mathbf{x}) = \text{constant}$. We write $p = p_c + \delta p$ to separate the bulk pressure from the pressure caused by local variations of $\sigma(\mathbf{x})$ and $c(\mathbf{x})$. These quantities are also separated into local and bulk parts, i.e., $c(\mathbf{x}) = c + \delta c(\mathbf{x})$ and $\sigma(\mathbf{x}) = \sigma + \delta\sigma(\mathbf{x})$. Canceling the bulk part we find that $\delta c = (\delta p / \delta\sigma - c) / (\sigma / \delta\sigma + 1)$. Because $\sigma / \delta\sigma \rightarrow 0$ as $T \rightarrow T_C$, the ratio $\delta p / \delta\sigma$ determines the near critical behavior of δc . $\delta p / \delta\sigma$ measures the uniform pressure change when a surface tension change occurs and it is clear that $\delta p \rightarrow 0$ as $\delta\sigma \rightarrow 0$ (i.e. $T \rightarrow T_C$). Near T_C the ratio $\delta p / \delta\sigma$ may either *i*) diverge, *ii*) converge to a constant, or *iii*) converge to zero. In case *i*) we find that $\delta c \sim \delta p (T_C - T)^{1-2\nu}$ as $T \rightarrow T_C$ so that the critical exponent for a curvature divergence would be weaker than $1-2\nu \approx -0.26$. Such a divergence of curvature probably occurred near the copper side-wall. We have seen something quite opposite in that the interface appears to flatten away from the side-wall in some parts of images F and G of Figure 3. This implies either that there is no curvature divergence (cases *ii*) and *iii*)) or there is no $\delta T(\mathbf{x})$ along the interface in this region. On the other hand, if $\delta p / \delta\sigma \rightarrow c_0 = \text{constant}$ (case *ii*) or $\delta p / \delta\sigma \rightarrow 0$ (case *iii*) as $T \rightarrow T_C$, then $c(\mathbf{x}) \rightarrow c_0$ or 0 as $T \rightarrow T_C$, i.e., a region of interface with a local change in temperature goes to this curvature value (case *ii*) or becomes flat (case *iii*)) near the critical point. In cases *ii*) and *iii*) a $\delta T(\mathbf{x})$ along the interface could help to explain these images. At $T \geq T_C$, the surface tension vanishes, the bubble's relaxation from surface tension is negligible, so that the “interface” shape is defined by local mass fluxes. In this case the interface evolution is analogous to the melting of a liquid-solid interface.

We next analyze another possible source of bubble deforming stress for an isothermal interface. The bubble may be deformed through the process of evaporation, i.e., by the normal stress exerted on the interface by the recoil from departing vapor [13,14]. Let n be the evaporating mass per unit time per unit interface area. The evaporating gas moves

normally to the interface, on average, and exerts a force per unit area (a « thrust ») on the liquid of $\delta p(\mathbf{x}) = n^2(\mathbf{x})(1/\rho_G - 1/\rho_L)$, where ρ denotes mass density and the subscripts L and G refer to liquid and gas respectively. In order to find the distribution $n(\mathbf{x})$ at the interface it is necessary to solve the entire heat transfer problem and this problem is complicated by the adiabatic heat transfer process. Because the temperature varies sharply in the boundary layer adjacent to the walls of the cell [10], the largest portion of mass transfer across the interface takes place near the triple contact line so that $n(\mathbf{x})$ is large in the vicinity of the contact line. A more detailed analysis [15] shows that $n(\mathbf{x})$ can exhibit a logarithmic divergence at the contact line decreasing exponentially far from it. We assume that $n(\mathbf{x})$ has the following form: $n(\mathbf{x}) = g(\mathbf{x})(T_C - T)^a$ as $T \rightarrow T_C$, i.e., it has the same local behavior with respect to temperature as the critical temperature is approached. The rate of change of mass of the vapor bubble is $dM/dt = \int n(\mathbf{x}) d\mathbf{x} = d/dt(V\phi\rho_G)$, where the integral is over the interfacial area, V is the cell volume, and ϕ is the constant vapor volume fraction (ϕ is the ratio of the gas volume to the total volume, $\phi = 1/2$ when the average density is the critical density ρ_c). Near the critical point the co-existence curve has the form $\rho_G = \rho_c - \Delta\rho/2$, where $\Delta\rho \sim (T_C - T)^\beta$ with $\beta = 0.325$, so that $dM/dt \sim (T_C - T)^{\beta-1} dT/dt$ as $T \rightarrow T_C$. Thus $a = \beta - 1$ and the curvature change due to the vapor recoil scales as $\delta c \sim \delta p / \sigma \sim (T_C - T)^{3\beta-2-2\nu}$. Because this critical exponent (≈ -2.3) is very large, it should manifest itself even far from the critical point in agreement with the experiments. This divergence is also much larger than the possible curvature divergence from a surface tension gradient. In summary, as $T \rightarrow T_C$, the vapor mass growth follows the growth of its density (the vapor volume remains constant), so that the diverging vapor production near the critical point drives a diverging recoil force.

The shape of the interface is governed by the equation $\delta p(\mathbf{x}) + p_c = c(\mathbf{x})\sigma$. Because c is proportional to the second derivative of the bubble shape, this governing formula is a

differential equation with the boundary condition given by the actual contact-angle. This actual contact-angle is the first derivative of the bubble shape function at the solid wall and is zero near the critical point. This problem can be reduced to 2-D as in the equilibrium case and we solved it using an expression for $\delta p(x)$ that contains the main physical features of the solution of the heat conduction problem [15] (the logarithmic divergence at the contact line and the rapid decay away from it). The influence of the vapor recoil force relative to the surface tension is measured using a non-dimensional parameter N , defined as $N = \int \delta p dl / \sigma$ where the integration is performed over the drop contour perpendicular to the contact line. Figure 5 shows how the apparent contact-angle increases with the increase of N . Because $N \sim (T_C - T)^{-2.3} \rightarrow \infty$ as $T \rightarrow T_C$, the N increase mimics the approach to the critical point and qualitatively explains the observed shape of the vapor bubble. The large apparent contact-angle can be understood by noting that the curvature increases sharply near the contact line. Because the interface slope changes so abruptly near the contact line, the contact-angle appears much larger than zero, as can be seen in Figure 5. In other experiments [16] under weightless conditions a similar drying process can be seen in the bubble images. Multiple bubble interactions and a small cell aspect ratio, however, complicated these experiments.

A very similar drying takes place during the liquid boiling process at large heat flux. When the heating to a surface is increased past a critical heat flux there is a sudden transition to "film" boiling, where the heater becomes covered with gas and may burnout [14, 15]. This "burnout" or "boiling crisis" is an important practical problem in many industries. We interpret the boiling crisis to be similar to the drying transition shown here [15]. Recent numerical calculations also support this interpretation [17]. The main difference is that the large value of N is made by a large vapor production that can be achieved during strong overheating rather than by the critical effects.

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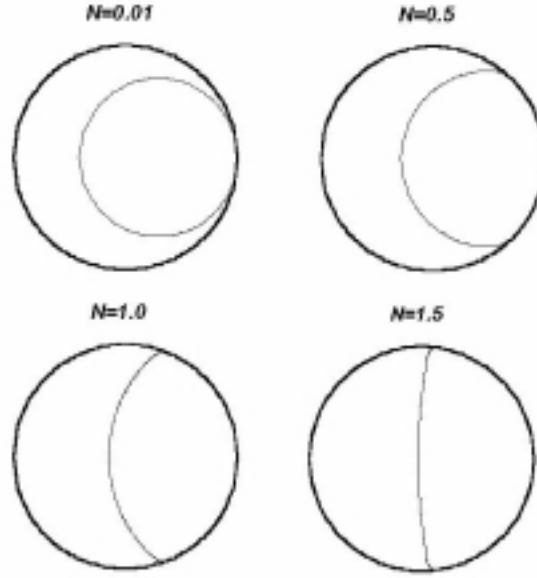


Figure 5. Calculated contact angle and bubble shape. The calculated shape of the vapor-liquid interface as described in the text for the different values of the non-dimensional strength of vapor recoil N that goes to infinity when the system approaches the critical point. Note that the actual contact angle is zero for all the curves.

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